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UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR COAL-TAR PITCH FOR ROOFING.

FEDERAL SPECIFICATIONS BOARD.

SPECIFICATION No. 80.

This specification was officially adopted by the Federal Specifications Board on December 29, 1923, for the use of the Departments and Independent Establishments of the Government in the purchase of coal-tar pitch for roofing.

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1. GENERAL.

This specification applies to coal-tar pitch intended for use with Coal-Tar Saturated Rag Felt for Roofing and Waterproofing (F. S. B. Spec. No. 81) in the construction of built-up roofing over board sheathing with inclines not exceeding 3 inches to the foot and over concrete surfaces with inclines not exceeding 1 inch per foot.

This coal-tar pitch when tested by the methods contained in this specification shall conform to the following requirements:

(a) APPEARANCE.—Freshly melted material shall have a uniform glossy black color, and on aging one week its surface shall not become dull or show any separation of oily constituents. Freshly fractured material shall present a satiny black surface.

(b) MELTING POINT (CUBE IN WATER).—140 to 150° F.

(c) DUCTILITY.—Minimum, 50 cm.

(d) SPECIFIC GRAVITY.—1.22 to 1.34.

(e) FREE CARBON.—15 to 30 per cent.

(f) DISTILLATION TEST.—Not more than 12 per cent by weight shall distill below 572° F. The specific gravity of the distillate shall be not less than 1.03.

(g) PACKING.—The coal-tar pitch shall be put up in steel drums, wooden barrels, or other suitable containers and shall be delivered at the site of the job in original sealed packages as put up by the manufacturer. The packages shall be marked with the manufacturer's name and brand.

When required, deliveries will, in general, be sampled and tested by the following methods, but the purchaser reserves the right to use any additional information to ascertain whether the material meets the specification.

2. SAMPLING.

From each shipment select at random a number of packages equivalent to the cube root of the total number of packages included in the lot. If the cube root as calculated proves to be a fractional number, express it as the next higher digit.

A sample shall be taken at least 3 inches below the surface and at least 3 inches from the side of each container selected. A clean hatchet may be used if the material is hard enough to shatter and a broad, stiff putty knife if the material is soft. An auger, or brace and three-fourths-inch bit, or other suitable means may also be used. Avoid contamination with superficial water, wooden slivers, or foreign matter.

The samples so taken shall be combined by melting together, with constant stirring to prevent overheating, at a temperature not exceeding 121° C. (250° F.). When thoroughly melted, two 1-quart friction-top cans shall be filled with the well-mixed material, allowed to cool, tightly sealed, marked for identification with the manufacturer's name and brand, etc., and one transmitted to the testing laboratory. The other shall be retained for use in case of dispute.

3. LABORATORY EXAMINATION.

(a) APPEARANCE.—Carefully melt about 10 g of the coal-tar pitch at a temperature not exceeding 121° C. (250° F.) and pour it into a small tin box or other suitable container provided with a cover. At least one-half the surface shall be free from froth or bubbles. Allow the coal-tar pitch to cool in a place free from drafts and dust. When cool, examine the surface, then cover the box with the lid and set aside for one week, after which period examine again. Immediately after cooling, the surface shall be

glossy black, and after standing one week the surface shall not show any dulling or the separation of greasy or oily material when compared with a freshly prepared sample.

(b) MELTING POINT (CUBE IN WATER METHOD).—The melting point shall be determined by the "Standard method of test for softening point of tar products (cube in water method)," A. S. T. M. Standards 1921, D 61-20. This method is essentially as follows:

Apparatus.—(See fig. 1). (1) A mold suitable for forming a 12.7 mm (one-half inch) cube of pitch. (2) An L-shaped hook made from No. 12 gauge copper wire, the foot of which shall be 2.54 cm (1 inch) long. (3) A beaker not less than 8.5 cm (3.3 inches) in diameter by 10.5 cm (4.1 inches) in depth measured from the bottom of the flare. (4) A thermometer which shall conform to the following specifications:

Total length, 370-400 mm (14.6-15.8 inches).

Diameter, 6.5-7.5 mm (0.26-0.30 inch).

Bulb length, not over 14 mm (not over 0.55 inch).

Bulb diameter, 4.5-5.5 mm (0.18 to 0.22 inch).

The scale shall be engraved upon the stem of the thermometer, shall be clear cut and distinct, and shall run from 0 to 80° C. (32 to 176° F.) in $\frac{1}{5}^{\circ}$ C. divisions. It shall commence at not less than 7.5 cm (3 inches) above the bottom of the bulb. The thermometer shall be correct to 0.25° C. (0.45° F.), as determined by comparison at full immersion with a similar thermometer calibrated at full immersion by the U. S. Bureau of Standards.

Preparation of sample.—The pitch shall be molded into a 12.7 mm (one-half inch) cube, truly shaped and with sharp edges, either by melting and pouring or softening and pressing into a mold. (Two satisfactory molds are shown in fig. 1.) In all cases an excess of pitch shall be used and the surplus material shall be cut off cleanly with a slightly heated knife. If the pitch is melted, it should be thoroughly stirred to avoid incorporating air bubbles in the mass and then poured into the mold so as to leave an excess on cooling. The mold should rest on a brass plate, and the surface of the plate and the interior surfaces of the mold should be amalgamated to prevent the pitch from adhering to them.

Procedure.—Assemble the apparatus as shown in Figure 1. Fill the beaker to a depth of approximately 9.5 cm ($3\frac{3}{4}$ inches) with freshly boiled distilled water at 15.5° C. (60° F.). Place the cube of pitch on the wire as shown in Figure 1 and suspend it in the water so that its lower edge is exactly 2.54 cm (1 inch) above

the bottom of the beaker and its upper edge is 5.1 cm (2 inches) below the surface of the water. Allow it to remain in the water for 15 minutes before applying heat. Suspend the thermometer so that the bottom of the bulb is level with the bottom edge of the cube of pitch and within 0.64 cm (one-fourth inch), but not touching the cube.

Apply the heat in such a manner that the temperature of the water is raised 5°C . (9°F .) each minute until the pitch softens and drops to the bottom of the beaker. The temperature recorded

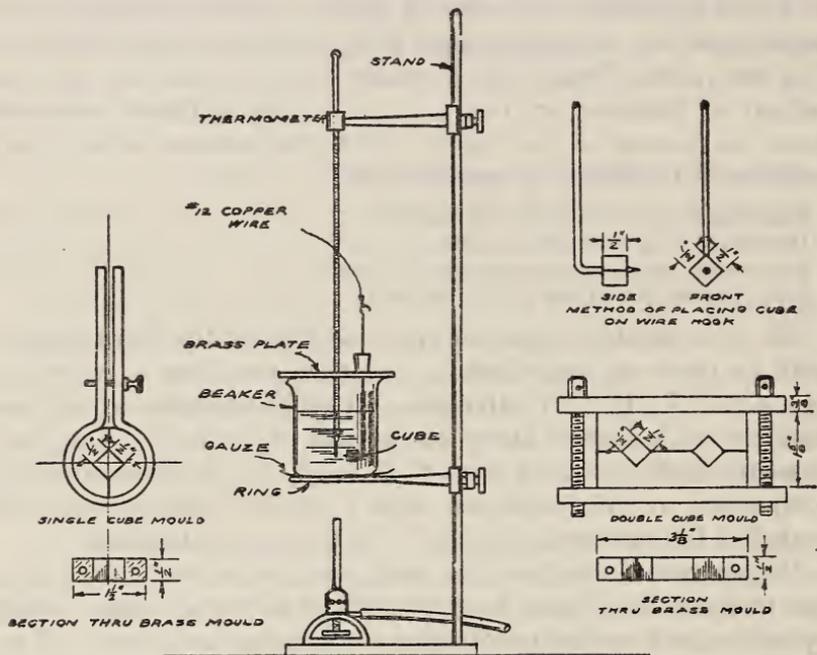


FIG. 1.—Apparatus for the determination of melting point.

by the thermometer at the instant the pitch touches the bottom of the beaker shall be taken as its melting point.

The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first three shall be $\pm 0.5^{\circ}\text{C}$. (0.9°F .) All tests in which the rate of rise in temperature exceeds these limits shall be rejected.

(c) DUCTILITY.—The ductility shall be determined by the "Tentative method of test for ductility of bituminous materials," D 113-22T, Proceedings A. S. T. M. 1922, vol. 22, page 807. This method is essentially as follows:

Apparatus.—Mold.—The mold shall be of the shape shown in Figure 2. It shall be made of brass, the ends *b* and *b'* being known as clips and the parts *a* and *a'* as sides of the mold. The dimensions of the mold shall be as follows:

	cm
Total length (internal).....	7.45-7.55
Distance between clips.....	2.97-3.06
Width of clips at mouth.....	1.98-2.02
Width of briquet at minimum cross section (halfway between clips).....	.99-1.01
Thickness of briquet throughout.....	.99-1.01

Water bath.—A water bath shall be maintained at a temperature varying not more than 0.1° C. from 25° C. (77° F.). The volume of water shall be not less than 10 liters, and the sample shall be

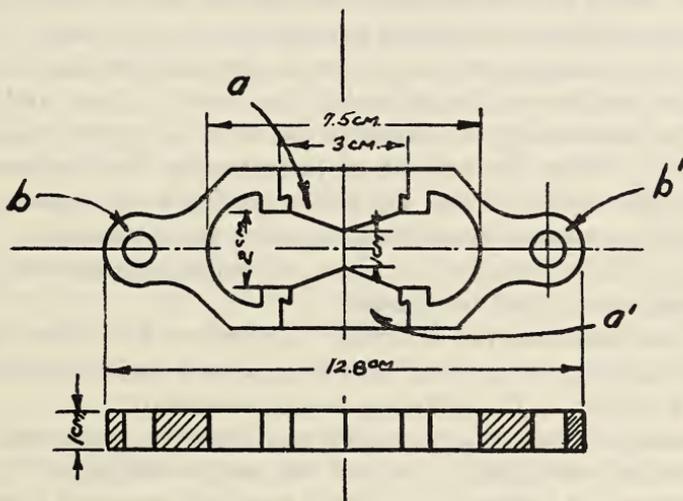


FIG. 2.—Dow ductility mold.

immersed to a depth of not less than 10 cm and shall be supported on a perforated shelf not less than 5 cm from the bottom of the bath.

Testing machine.—Any apparatus may be used for pulling the briquets of pitch apart which is so constructed that the briquet will be continuously immersed in water and the two clips pulled apart at a uniform rate of 5 cm per minute.

Procedure.—The coal-tar pitch shall be completely melted at the lowest temperature at which it will be thoroughly fluid and then poured into the standard mold. The mold shall be assembled on a brass plate, and the surface of the plate and the interior surfaces of the side pieces *a* and *a'* of the mold shall be thoroughly amalgamated so as to prevent the pitch from sticking. In filling the mold care shall be taken not to disarrange the parts

and thus distort the briquet. In filling, the pitch shall be poured in a thin stream back and forth from end to end of the mold until it is more than level full. After cooling to room temperature for 30 minutes the excess pitch shall be cut off by means of a hot putty knife, so that the mold will be just level full.

The brass plate and mold containing the briquet shall then be placed in the water bath and kept at a temperature of 25° C. (77° F.) for at least one and one-half hours, when the briquet shall be removed from the plate and the side pieces, *a* and *a'*, detached.

The rings at each end of the clip shall then be attached to the pins or hooks in the ductility machine and the two clips pulled apart at a uniform rate of 5 cm per minute (± 5 per cent). While the test is being made, the water in the tank of the ductility machine shall cover the briquet by at least 2.5 cm and shall be kept continuously at a temperature of 25° C. (77° F.) $\pm 0.5^\circ$ C. (0.9° F.). When the briquet of pitch breaks, the distance from the original position of the clip before pulling to its present position shall be measured and shall be taken as the ductility. The average of three tests shall be taken, excepting that any obviously abnormal result shall be rejected.

(*d*) SPECIFIC GRAVITY AT 25°/25° C. (77°/77° F.).—The specific gravity may be determined by any approved method, but some method similar to the following is recommended:

A small platinum pan or crucible supported in a wire sling shall be accurately weighed in air and the weight designated by (*a*). It shall then be weighed in a similar manner suspended in freshly distilled water at 25° C., but immersed except for the fine platinum wire used for suspension and this weight designated by (*b*). The crucible shall then be dried and poured almost full of coal-tar pitch which has been melted at the lowest possible temperature. It shall then be allowed to cool to 25° C. (77° F.) and weighed in the sling in air. This weight shall be designated as (*c*). Then it shall be weighed in water, and the weight shall be designated as (*d*). From these weights the specific gravity shall be computed according to the following formula:

$$\text{Sp. gr.} = \frac{c - a}{(c - a) - (d - b)}$$

(*e*) FREE CARBON.—Free carbon, as defined by the American Society for Testing Materials, is that matter in tars minus the ash which is insoluble in cold carbon bisulphide.

Method No. 1.—One g of coal-tar pitch shall be placed in a beaker of 100 to 150 cc capacity, covered with cold carbon bisulphide and the whole stirred every few minutes for about 15 minutes, until the lumps break up and the bitumen goes into solution. The solid particles shall be allowed to settle, and the carbon bisulphide solution of the bitumen shall be decanted through a weighed Gooch crucible, prepared with a mat of asbestos fiber of a thickness that will scarcely transmit light.

The sediment remaining in the beaker shall be crushed with a stirring rod and a little more carbon bisulphide added. The sides of the beaker also shall be washed down with carbon bisulphide. The contents of the beaker shall then be poured into the crucible and all sediment washed from the beaker into the crucible with carbon bisulphide. Suction may be used to aid in filtering. The sediment on the filter shall be washed with carbon bisulphide until the washings run through colorless. The crucible shall then be set in a place free from dust until the carbon bisulphide evaporates and then placed in an oven at 105° C. (221° F.) for one hour. It shall then be cooled and weighed, the increase giving the total insoluble matter.

To determine the ash, 1 g of the pitch shall be weighed in a crucible of about 30 cc capacity and incinerated until the carbon is consumed and only mineral matter remains. Cool and weigh, and compute the ash.

Subtract the amount of ash from the total insoluble matter and consider the difference as free carbon. Should the ash exceed 1 per cent, it is an indication that mineral matter has been added to the pitch and the material should be rejected. Comparative results may be obtained by the following method, but in case of dispute the method given above will be used.

Method No. 2.—A 1 g sample of the coal-tar pitch shall be placed in a 100 cc beaker and digested on a steam bath with about 50 cc of c. p. toluol for not over 20 minutes and the liquid stirred occasionally to break up lumps and solid particles. An extraction thimble or filter cup shall be prepared by washing with hot benzol and drying at 105° C. (221° F.) for one hour. It shall be cooled in a desiccator and weighed in a weighing bottle. The extraction thimble in a glass filter cup shall be held over a beaker and wet with c. p. toluol, then the toluol-pitch solution shall be decanted through it. Both it and the beaker shall be washed with hot c. p. toluol until clean, particles adhering to the beaker being detached with a camel's-hair brush. The extraction thim-

ble shall then be washed with c. p. benzol, allowed to drain, covered with a cap of filter paper, placed in the extraction apparatus, and extracted with benzol until the descending benzol runs through colorless. The thimble shall then be removed, the cap taken off, and the thimble dried at 105° C. (221° F.) for one-half hour. It shall then be placed in the weighing bottle, cooled in a desiccator, and weighed. The increase in weight is total insoluble matter. Determine the ash as in method No. 1 and subtract it from the total insoluble matter to obtain the amount of free carbon.

(f) **DISTILLATION TEST.**—The coal-tar pitch shall be distilled by the "Standard method of test for distillation of bituminous materials for road treatment," D 20-18, A. S. T. M. Standards, 1921, page 735. This method is essentially as follows:

Apparatus.—The apparatus shall consist of the following (see fig. 3):

(1) *Flask.*—A 250 cc Engler distilling flask, having the following dimensions:

Diameter of bulb.....	8.0 cm
Length of neck.....	15.0 cm
Diameter of neck.....	1.7 cm
Surface of pitch to lower side of tubulature.....	11.0 cm
Length of tubulature.....	15.0 cm
Diameter of tubulature.....	.9 cm
Angle of tubulature.....	75°

A variation of 3 per cent from the above measurements is permissible.

(2) *Thermometer.*—An A. S. T. M. distillation thermometer conforming to the following specifications:

Type.—Jena or Corning glass thoroughly annealed.

Total length.—Maximum, 385 mm.

Stem.—Diameter, 7 mm; permissible variation, 0.5 mm.

Bulb.—Length, 12.5 mm; permissible variation, 2.5 mm; diameter, minimum, 5 mm, and shall not exceed diameter of stem.

Actuating liquid.—Mercury.

Range.—0 to 400° C.

Immersion.—Total.

Distance to 0° C. mark from bottom of bulb.—30 mm; permissible variation, 5 mm.

Distance from 0 to 400° C. mark.—295 mm; permissible variation, 10 mm.

Filling.—Nitrogen gas.

Reservoir.—Above final graduation large enough so that pressure will not become excessive at highest temperature.

Top finish.—Small glass ring or button.

Graduation.— 0° to 400° C. in 1° C. intervals. Each fifth graduation shall be longer than the intermediate ones, and every tenth graduation beginning at zero shall be numbered.

Special markings.—A. S. T. M. distillation, serial number, and manufacturer's name.

Accuracy.—Maximum error between following points:

From—

0 to 200° C.	0.5° C.
200 to 300° C.	1.0° C.
300 to 375° C.	1.5° C.

Sensitiveness.—When cooled to a temperature of 74° C. below the boiling point of water at barometric pressure at the time of test and plunged into a free flow of stream, the meniscus shall pass the point 10° C. below the boiling point of water in not more than six seconds.

Tests for permanency.—The thermometer shall be set up as for the distillation test, water, naphthalene, and benzophenone being used as distilling liquids. The correctness of the thermometer shall be checked at 0 and 100° C. after each third distillation until seasoned.

(3) *Condenser.*—The condenser tube shall have the following dimensions:

	mm
Adapter.	70
Length of straight tube.	185
Width of tube.	12-15
Width of adapter end of tube.	20-25

(4) *Stands.*—Two iron stands—one with a universal clamp for holding the condenser and one with a light grip arm with a cork-lined clamp for holding the flask.

(5) *Burner and shield.*—A Bunsen burner and a tin shield 20 cm long by 9 cm in diameter. The shield shall have a small hole for observing the flame.

(6) *Cylinder.*—The cylinder used in collecting the distillate shall have a capacity of 25 cc and shall be graduated in 0.1 cc.

Procedure.—Assemble the apparatus as in Figure 3, the thermometer being placed so that the top of the bulb is opposite the middle of the tubulature. All connections should be tight.

One hundred g of the pitch shall be placed in the flask and distilled at the uniform rate of 1 cc per minute. The distillate shall be collected in a graduate and the distillation stopped on

reaching 300° C. (572° F.). Weigh the distillate. During the distillation the condenser tube shall be warmed when necessary to prevent the deposition of any sublimate.

(g) SPECIFIC GRAVITY OF DISTILLATE.—A specific gravity bottle or pycnometer shall be cleaned, dried at 105° C. (221° F.), cooled, and weighed. It shall then be filled with freshly boiled distilled water at 38° C. (100° F.) and weighed. The bottle shall then be dried and filled at 38° C. (100° F.) with distillate obtained in 3 (f)

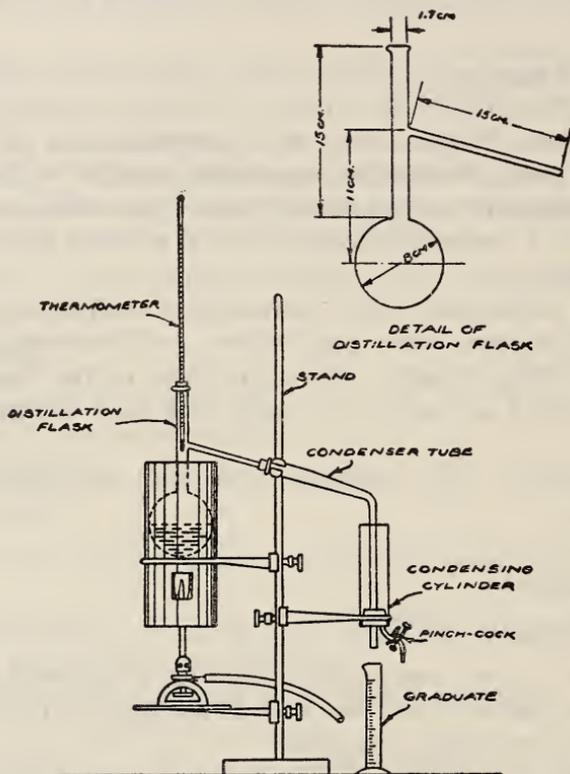


FIG. 3.—Distillation apparatus.

and weighed. From these weights the specific gravity can be computed according to the following formula:

$$\text{Sp. gr. at } 38^{\circ}/15.5^{\circ} \text{ C. (} 100^{\circ}/60^{\circ} \text{ F.)} = \frac{c-a}{b-a} \times \frac{0.99299}{0.99913}$$

a = weight of bottle

b = weight of bottle and distilled water

c = weight of bottle and distillate

0.99299 = density of water at 38° C.

0.99913 = density of water at 15.5° C.

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